5,7,7-Trimethyloctannitril

[0001] The present invention relates to 5,7,7-trimethyloctanenitrile, certain products containing a proportion of said substance (in particular fragrance compositions, perfumed articles and bleach compositions) and certain uses of the substance.

[0002] Despite a multiplicity of already available fragrances, there continues to be a general need in the perfume industry for new fragrances which, in addition to their primary, specific odour, properties, have additional positive secondary properties such as, for example a higher stability under certain application conditions, improved spreading capacity, a better adhesion or the like.

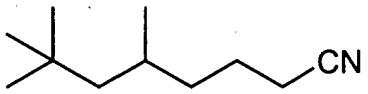
[0003] It has already been known for a long time that the chemical group of substances comprising the nitriles contains a number of interesting fragrances. Known commercial products are citronellyl cyanide, geranyl cyanide and cinnamyl cyanide. A review of nitriles as fragrances is given by Vasanti G. Yadav in Perfumery Nitriles and Acetals: Part II, Synthesis and Characteristics of Nitriles, PAFAI J. (1994), 16 (2), 29 - 42. More recent publications on nitriles as constituents of perfumes are the patent documents US 5,179, 222, US 6,180,814 and US 5,521, 151. The latter documents relate to nitriles, which, in addition to their primary odour characteristic, are characterized by a particularly high stability in aggressive media (for example aqueous solutions having a particularly high or particularly low pH value).

[0004] The primary aim of the present invention was to indicate a chemical compound, which can be used as a fragrance with a

pronounced iris note and also has a high stability in aggressive media.

[0005] According to the invention, this aim is achieved by indicating the compound 5,7,7-trimethyloctanenitrile. In the context of the present text, the term 5,7,7-trimethyloctanenitrile includes the R-configured enantiomers, the S-configured enantiomers and (in particular racemic) mixtures of the enantiomers.

[0006] The structural formula of 5,7,7-trimethyloctanenitrile



is given below:

[0007] The odour characteristics of 5,7,7-trimethyloctanenitrile are described as: iris, vetiver, iris butter, woody, rooty.

[0008] In addition to its excellent odour characteristics, the substance also has an excellent stability in aggressive media, in which context bleach systems (bleaching agents) may be mentioned here in particular.

[0009] Surprisingly, it has been found that in respect of its properties, the invention (sic) according to the invention 5,7,7-trimethyloctanenitrile is even superior to the compound 2-nonenenitrile $CH_2(CH_2)CH=CH-CN$ which is also known by the name "iris nitrile". This is because 2-nonenenitrile has an odour strength that is undesired in perfumery, which makes it such a penetrating fragrance that it is justifiably stated in Arctander S., "Perfume and Flavour Chemicals", Vol. I, 1969, No. 2362, that the main difficulty in promoting this substance as a fragrance lies in its enormous (odour) strength. 5,7,7-

trimethyloctanenitrile according to the invention is, in contrast, less penetrating and also differs from 2-nonenenitrile in respect of the odiferous secondary aspects, which are particularly appreciated by perfumerers.

[00010] As supplementary information, it is pointed out that in the tabular review of the odour characteristics of synthetic nitriles, which contains 48 substances, that is given by Yadav loc. cit., there is only one, specifically the compound 3-methyl-2-nonenenitrile (compound 13), that is designated as a fragrance having an iris note.

[00011] Whilst nitriles frequently have an odour that corresponds to that of the corresponding aldehydes (again cf. Yadav loc. cit.), there is no such odour relationship between 5,7,7-trimethyloctanenitrile according to the invention and its corresponding aldehyde. Thus, there is an odour deviation between nitrile and aldehyde, which Yadav would have termed an "anomalous odour effect".

[00012] The present invention also relates to fragrance compositions and to perfumed articles that contain an amount of 5,7,7-trimethyloctanenitrile that is effective from the sensory standpoint. Perfumerers who have worked on the use of the substance according to the invention have found that this has an (even) better performance than iris nitrile as a fragrance since, compared with iris nitrile, it produces more performance when used in a smaller amount.

[00013] The amount of 5,7,7-trimethyloctanenitrile in a fragrance composition according to the invention is preferably sufficient to modify and/or to intensify the fragrance composition in the iris direction.

[00014] According to the invention, a fragrance composition with an iris note is prepared by mixing 5,7,7-trimethyloctanenitrile

with conventional further constituents of a fragrance composition, the 5,7,7-trimethyloctanenitrile being used in an amount that is sufficient to modify and/or to intensify the odour of the fragrance composition in the iris direction.

[00015] The use of the substance according to the invention in a bleaching agent composition is particularly preferred. Accordingly, a bleaching agent composition according to the invention contains

a bleaching agent,

5,7,7-trimethyloctanenitrile and, optionally,

conventional additives,

the 5,7,7-trimethyloctanenitrile being present in an amount that is sufficient to modify and/or to intensify the odour of the bleaching agent composition in the iris direction. In this context, it is preferred that the bleaching agent comprises chlorine and/or hypochlorite.

[00016] Further preferred aspects and sub-aspects of the invention result from the following examples and the appended patent claims:

Examples:

Perfume compositions:

[00017] Example of the use of 5,7,7-trimethyloctanenitrile:

Aldehyde C-10 1 % DPG	8.00
Aldehyde C-11 Lenic 1 % DPG	6.00
Aldehyde C-9 10 % DPG	4.00
Benzyl acetate	65.00
Brahmanol [1)]	10.00
Cinnamic alcohol	20.00
Citronellol inactive	90.00
Citronellyl acetate	40.00
Clary sage oil Russia 10 % DPG	6.00
DPG (dipropylene glycol)	132.00
Dupical [2)] 10 % DPG	3.00
Exaltolide Total [3)]	2.00

Hedione [4)]	8.00
Hexenyl acetate cis-3 10 % DPG	8.00
Hexylcinnamic aldehyde	160.00
Indole 10 % DPG	15.00
Indolene 50 [5)]	10.00
Lemon Oil Italian SFUmatrice	2.00
Lilial [6)]	20.00
Linalool synth.	90.00
Lyral [7)]	50.00
Phenylethyl acetate	25.00
Phenylethyl alcohol	140.00
Phenylethyl isobutyrate	5.00
Rose oxide l 1 % DPG	6.00
Sandalwood oil East India	5.00
Terpineol pure	60.00
5,7,7-Trimethyloctanenitrile	10.00
	1000.00

Trade name owned by Dragoco, Holzminden, D
Trade name owned by Quest, Ashford, GB
3),4) Trade name owned by Firmenich, Geneva, CH
5),6) Trade name owned by Givaudan, Zürich, CH
Trade name owned by IFF, New Jersey, US

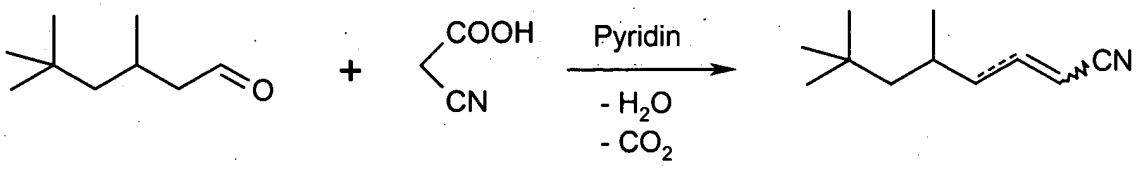
[00018] Description of the odour: flowery, lily of the valley, very natural, very soft, iris.

[00019] Bleaching agent composition:

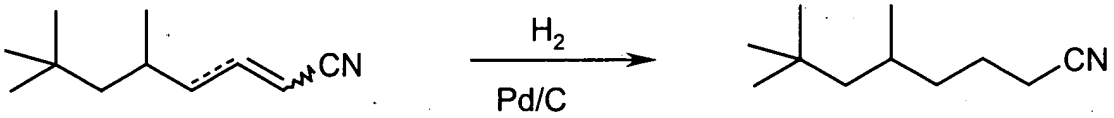
Sodium laurate (as 30 % solution)	1.5
Coconut stearyldimethylamine oxide	3.5
Hypochlorite (as 13 % solution)	40.0
Sodium hydroxide (as 30 % solution)	2.5
Water	52.4
5,7,7-Trimethyloctanenitrile	0.1
	100.0

Preparation:

[00020] The preparation of 5,7,7-trimethyloctanenitrile is carried out in a manner known per se. Thus, Z/E-5,7,7-trimethyl-2(3)-octenenitrile which, surprisingly, from the odour standpoint is to be characterised completely differently to the compound according to the invention, cf. Yadov loc. cit., compound 8, can advantageously be prepared from isononyl aldehyde and cyanoacetic acid by means of a Knoevenagel reaction.



[00021] The subsequent hydrogenation to give the saturated



nitrile is then advantageously carried out using Pd/C:

[00022] Method of preparation:

[00023] Step 1: Preparation of E/Z-5,7,7-trimethyl-2(3)-octenenitrile

300g Toluene, 255 g (3 mol) cyanoacetic acid and 426 g (3 mol) isononyl aldehyde are initially introduced in the indicated order and the mixture is heated to 55-60°C with stirring. 64 g Pyridine is then added dropwise in the course of 15 minutes without further heating. The temperature rises to 65°C. The mixture is then heated to the boil in the course of 45 min, and the water of reaction is separated off for 4 h in a water separator. The reaction mixture is cooled to 70-75°C and washed at 70°C with 2× 150 g water, 2× 150 g 10 % H_2SO_4 and 1× 150 g NaCl solution. It is then distilled.

[00024] Yield of E/Z-5,7,7-trimethyl-2(3)-octenenitrile (4 isomers): 270.7 g (55 %)

[00025] Step 2: Hydrogenation to give 5,7,7-trimethyloctanenitrile

270 g (1.6 mol) E/Z-5,7,7-trimethyl-2(3)-octenenitrile, 1.8 l ethanol and 3.24 g Pd/C are stirred for 10 h at 30°C and 40 bar under hydrogen. GC is used to check that the hydrogenation has gone to completion.

[00026] The solvent is then stripped off and fractionation is carried out on a 20 cm GFCC.

[00027] Yield: 210.1 g (77 %).

Spectroscopic data:

[00028] 5,7,7-Trimethyloctanenitrile:

^{1}H-NMR ($CDCl_3$, 300 MHz, TMS= 0 ppm): δ = 0.90 (s, 9 H); 0.93 (d, 3 H, *J*= 6.5 Hz); 1.08 (dd, 1 H, *J*= 14.0 and 6.2 Hz); 1.21 (dd, *J*= 14,0 and 3.5 Hz, 1 H); 1.25 - 1.55 (m, 3 H); 1.55 - 1.72 (m, 2 H); 2.32 (t, 2 H, *J*= 7,14 Hz).

[00029] ^{13}C-NMR ($CDCl_3$, 75 MHz): δ = 17.4 (t); 22.4 (q); 23.2 (t); 28.7 (d); 30.0 (3 q); 31.0 (s); 38.4 (t); 51.0 (t); 119.8 (s).

[00030] MS (m/e, %): 166 (M^+,0); 152 (58); 110 (13); 96 (23); 69 (13); 57 (100); 56 (11); 55 (13); 41 (26).

Precursor E/Z-5,7,7-trimethyl-2(3)-octenenitrile:

[00031] MS (m/e, %):

Isomer 1: 165 (M,0); 150 (60); 108 (20); 94 (23); 67 (40); 57 (100); 41 (38).

Isomer 2: 165 (M,1); 150 (20); 109 (25); 94 (50); 71 (40); 57 (100); 41 (30).

Isomer 3: 165 (M,0); 150 (20); 123 (20); 108 (20); 99 (25); 67 (30); 57 (100); 41 (30).

Isomer 4: 165 (M,15); 150 (20); 109 (40); 94 (15); 71 (20); 57 (100); 41 (40).

[00032] IR:

Isomer 1: ν (cm^{-1}) = 2963.21; 2915.05; 1472.96; 1371.26; 1241.54.

Isomer 2: ν (cm^{-1}) = 2964.67; 2914.47; 1471.77; 1371.36.

Isomer 3: ν (cm^{-1}) = 2963.98; 2918.76; 2228.71; 1634.35; 1473.54; 1371.04; 966.55.

Isomer 4: ν (cm^{-1}) = 2964.57; 2916.06; 1472.04; 1370.03; 971.37.